

November 10, 2016

Tom Moe  
USS Corporation  
P.O. Box 417  
8771 Park Ridge Dr  
Mountain Iron, MN 55768

RE: Project: USS MinnTac NPDES-TB Wk1  
Pace Project No.: 1278234

Dear Tom Moe:

Enclosed are the analytical results for sample(s) received by the laboratory on November 02, 2016. The results relate only to the samples included in this report. Results reported herein conform to the most current, applicable TNI/NELAC standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,



Melisa M Woods  
melisa.woods@pacelabs.com  
Project Manager

Enclosures

cc: Cory Hertling  
Terri Sabetti, NTS



## REPORT OF LABORATORY ANALYSIS

This report shall not be reproduced, except in full,  
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## CERTIFICATIONS

Project: USS MinnTac NPDES-TB Wk1

Pace Project No.: 1278234

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### Virginia Minnesota Certification ID's

315 Chestnut Street, Virginia, MN 55792

Alaska Certification UST-107

Alaska Certification UST-107

Alaska Certification #MN01084

Arizona Department of Health Certification #AZ0785

Minnesota Dept of Health Certification #: 027-137-445

North Dakota Certification: # R-203

Wisconsin DNR Certification # : 998027470

WA Department of Ecology Lab ID# C1007

Nevada DNR #MN010842015-1

Oklahoma Department of Environmental Quality

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### Duluth Minnesota Certification ID's

4730 Oneota St., Duluth, MN 55807

Minnesota Dept of Health Certification #: 027-137-152

Wisconsin DNR Certification # : 999446800

North Dakota Certification #: R-105

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## REPORT OF LABORATORY ANALYSIS

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## SAMPLE SUMMARY

Project: USS MinnTac NPDES-TB Wk1

Pace Project No.: 1278234

Lab ID	Sample ID	Matrix	Date Collected	Date Received
1278234001	SD 001 (Seep 020)	Water	11/02/16 10:30	11/02/16 13:25

## REPORT OF LABORATORY ANALYSIS

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## SAMPLE ANALYTE COUNT

Project: USS MinnTac NPDES-TB Wk1

Pace Project No.: 1278234

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
1278234001	SD 001 (Seep 020)	EPA 1664A TPH (1999)	DES	1	PASI-DUL
		USGS I-3765	JJH	1	PASI-V
		EPA 300.0	DMB	1	PASI-V

## REPORT OF LABORATORY ANALYSIS

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## ANALYTICAL RESULTS

Project: USS MinnTac NPDES-TB Wk1

Pace Project No.: 1278234

Sample: SD 001 (Seep 020)		Lab ID: 1278234001		Collected: 11/02/16 10:30		Received: 11/02/16 13:25		Matrix: Water	
Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
<b>1664 SGT-HEM, TPH</b>									
Analytical Method: EPA 1664A TPH (1999)									
Total Petroleum Hydrocarbons	ND	mg/L	3.0	1.0	1		11/07/16 10:05		
<b>USGS I-3765 TSS</b>									
Analytical Method: USGS I-3765									
Total Suspended Solids	ND	mg/L	1.0	1.0	1		11/07/16 14:53		
<b>300.0 IC Anions 28 Days</b>									
Analytical Method: EPA 300.0									
Sulfate	914	mg/L	20.0	10.0	10		11/10/16 08:02	14808-79-8	

## REPORT OF LABORATORY ANALYSIS

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## QUALITY CONTROL DATA

Project: USS MinnTac NPDES-TB Wk1

Pace Project No.: 1278234

QC Batch:	99367	Analysis Method:	EPA 1664A TPH (1999)
QC Batch Method:	EPA 1664A TPH (1999)	Analysis Description:	1664 SGT-HEM, TPH
Associated Lab Samples:	1278234001		

METHOD BLANK: 394453 Matrix: Water

Associated Lab Samples: 1278234001

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Total Petroleum Hydrocarbons	mg/L	ND	3.0	1.0	11/07/16 10:05	

LABORATORY CONTROL SAMPLE: 394454

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Total Petroleum Hydrocarbons	mg/L	20	17.4	87	64-132	

MATRIX SPIKE SAMPLE: 394455

Parameter	Units	1278127001 Result	Spike Conc.	MS Result	MS % Rec	% Rec Limits	Qualifiers
Total Petroleum Hydrocarbons	mg/L	1.2J	20	17.8	83	64-132	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

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## QUALITY CONTROL DATA

Project: USS MinnTac NPDES-TB Wk1

Pace Project No.: 1278234

QC Batch: 99414

Analysis Method: USGS I-3765

QC Batch Method: USGS I-3765

Analysis Description: USGS I-3765 Total Suspended Solids

Associated Lab Samples: 1278234001

METHOD BLANK: 394655

Matrix: Water

Associated Lab Samples: 1278234001

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Total Suspended Solids	mg/L	ND	1.0	1.0	11/07/16 14:52	

LABORATORY CONTROL SAMPLE: 394656

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Total Suspended Solids	mg/L	239	236	99	80-120	

SAMPLE DUPLICATE: 394657

Parameter	Units	1278456001 Result	Dup Result	RPD	Max RPD	Qualifiers
Total Suspended Solids	mg/L	90.0	87.5	3	10	

SAMPLE DUPLICATE: 394658

Parameter	Units	1278412002 Result	Dup Result	RPD	Max RPD	Qualifiers
Total Suspended Solids	mg/L	20.0	20.0	0	10	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

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## QUALITY CONTROL DATA

Project: USS MinnTac NPDES-TB Wk1

Pace Project No.: 1278234

QC Batch: 99654

Analysis Method: EPA 300.0

QC Batch Method: EPA 300.0

Analysis Description: 300.0 IC Anions

Associated Lab Samples: 1278234001

METHOD BLANK: 395644

Matrix: Water

Associated Lab Samples: 1278234001

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Sulfate	mg/L	ND	2.0	1.0	11/09/16 18:38	

LABORATORY CONTROL SAMPLE: 395645

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Sulfate	mg/L	50	48.1	96	90-110	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 395646

395647

Parameter	Units	1278306001 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Sulfate	mg/L	<1.0	50	50	48.9	48.8	98	98	90-110	0	20	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 395648

395649

Parameter	Units	1278412002 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Sulfate	mg/L	ND	500	500	492	488	97	97	90-110	1	20	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

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## QUALIFIERS

Project: USS MinnTac NPDES-TB Wk1  
Pace Project No.: 1278234

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### DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

### LABORATORIES

PASI-DUL Pace Analytical Services - Duluth

PASI-V Pace Analytical Services - Virginia

## REPORT OF LABORATORY ANALYSIS

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## QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: USS MinnTac NPDES-TB Wk1


Pace Project No.: 1278234

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
1278234001	SD 001 (Seep 020)	EPA 1664A TPH (1999)	99367		
1278234001	SD 001 (Seep 020)	USGS I-3765	99414		
1278234001	SD 001 (Seep 020)	EPA 300.0	99654		

## REPORT OF LABORATORY ANALYSIS

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	Document Name: Sample Condition Upon Receipt Form	Document Revised: 23Feb2015 Page 1 of 1
	Document No.: F-VM-C-001-Rev.09	Issuing Authority: Pace Virginia, Minnesota Quality Office

Sample Condition  
Upon Receipt

Client Name:

NSS

Project #:

WO#: 1278234



Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☒ Client  
☐ Commercial ☐ Pace ☐ Other: \_\_\_\_\_

Tracking Number: \_\_\_\_\_

Custody Seal on Cooler/Box Present? ☐ Yes ☒ No Seals Intact? ☐ Yes ☒ No

Optional: Proj. Due Date: \_\_\_\_\_ Proj. Name: \_\_\_\_\_

Packing Material: ☐ Bubble Wrap ☒ Bubble Bags ☐ None ☐ Other: \_\_\_\_\_ Temp Blank? ☒ Yes ☐ No

Thermometer Used: ☒ 140792808 Type of Ice: ☒ Wet ☐ Blue ☐ None ☒ Samples on ice, cooling process has begun

Cooler Temp Read °C: 2.8 Cooler Temp Corrected °C: 3.1 Biological Tissue Frozen? ☐ Yes ☐ No ☒ NA

Temp should be above freezing to 6°C Correction Factor: +0.3 Date and Initials of Person Examining Contents: CR 11-2-16

Comments:

Chain of Custody Present?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	1.
Chain of Custody Filled Out?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	2.
Chain of Custody Relinquished?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	3.
Sampler Name and Signature on COC?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	4.
Samples Arrived within Hold Time?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	5.
Short Hold Time Analysis (<72 hr)?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	6.
Rush Turn Around Time Requested?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	7.
Sufficient Volume?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	8.
Correct Containers Used?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	9.
-Pace Containers Used?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	
Containers Intact?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	10.
Filtered Volume Received for Dissolved Tests?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	11. Note if sediment is visible in the dissolved containers.
Sample Labels Match COC?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	12.
-Includes Date/Time/ID/Analysis Matrix: <u>WT</u>		
All containers needing acid/base preservation will be checked and documented in the pH logbook.	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	See pH log for results and additional preservation documentation
Headspace in Methyl Mercury Container	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	13.
Headspace in VOA Vials (>6mm)?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	14.
Trip Blank Present?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	15.
Trip Blank Custody Seals Present?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	
Pace Trip Blank Lot # (if purchased): _____		

CLIENT NOTIFICATION/RESOLUTION

Field Data Required? ☐ Yes ☐ No

Person Contacted: \_\_\_\_\_ Date/Time: \_\_\_\_\_

Comments/Resolution: \_\_\_\_\_

FECAL WAIVER ON FILE Y N

TEMPERATURE WAIVER ON FILE Y N

Project Manager Review:

*[Signature]*

Date: 11/2/16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e. out of hold, incorrect preservative, out of temp, incorrect containers)

# Intra-Regional Chain of Custody



Workorder: 1278234      Workorder Name: NPDES-TB WK1      Owner Received Date: 11/2/2016      Due Date: 11/16/2016


Received at: **Pace Analytical Virginia**  
315 Chestnut Street  
Virginia, MN 55792  
Phone (218) 742-1042

Send To Lab: **Pace Analytical Duluth**  
4730 Oneota Street  
Duluth, MN 55807  
Phone (218) 727-6380

Report To:  
Melisa M Woods

Item	Sample ID	Sample Type	Collect Date/Time	Lab ID	Matrix	HCL	Preserved Containers	EPA 1661A TPH (1999)	LAB USE ONLY																														
1	SD 001 (Seep 020)	PS	11/2/2016 10:30	1278234001	Water	3		X																															
2																																							
3																																							
4																																							
5																																							
<table border="1"> <thead> <tr> <th>Transfers</th> <th>Released By</th> <th>Date/Time</th> <th>Received By</th> <th>Date/Time</th> <th>Comments</th> </tr> </thead> <tbody> <tr> <td>1</td> <td>CCCP</td> <td>11/2/16 14c</td> <td>[Signature]</td> <td>11/3/16 14c</td> <td></td> </tr> <tr> <td>2</td> <td>[Signature]</td> <td>11/3/16 1530</td> <td>[Signature]</td> <td>11/3/16 1530</td> <td></td> </tr> <tr> <td>3</td> <td></td> <td></td> <td></td> <td></td> <td></td> </tr> <tr> <td>4</td> <td></td> <td></td> <td></td> <td></td> <td></td> </tr> </tbody> </table>										Transfers	Released By	Date/Time	Received By	Date/Time	Comments	1	CCCP	11/2/16 14c	[Signature]	11/3/16 14c		2	[Signature]	11/3/16 1530	[Signature]	11/3/16 1530		3						4					
Transfers	Released By	Date/Time	Received By	Date/Time	Comments																																		
1	CCCP	11/2/16 14c	[Signature]	11/3/16 14c																																			
2	[Signature]	11/3/16 1530	[Signature]	11/3/16 1530																																			
3																																							
4																																							
Cooler Temperature on Receipt <u>0.4</u> °C      Custody Seal <u>Y</u> or N      Received on Ice <u>Y</u> or N      Samples Intact <u>Y</u> or N																																							

\*\*\*In order to maintain client confidentiality, location/name of the sampling site, sampler's name and signature may not be provided on this COC document.  
This chain of custody is considered complete as is since this information is available in the owner laboratory.

	Document Name: Sample Condition Upon Receipt Form	Document Revised: 22Jan2016 Page 1 of 1
	Document No.: F-DUL-C-001-Rev.01	Issuing Authority: Pace Virginia, Minnesota Quality Office

Sample Condition  
Upon Receipt

Client Name:

Project #:

IR COC VIRG.

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client  
☐ Commercial ☒ Pace ☐ Other: \_\_\_\_\_

Tracking Number: \_\_\_\_\_

Custody Seal on Cooler/Box Present?

☒ Yes ☐ No

Seals Intact?

☒ Yes ☐ No

Optional: Proj. Due Date: Proj. Name:

Shipping Material: ☒ Bubble Wrap

☐ Bubble Bags

☐ None

☐ Other: \_\_\_\_\_

Temp Blank? ☒ Yes ☐ No

Thermometer Used: ☒ 800051

Type of Ice: ☒ Wet

☐ Blue

☐ None

☒ Samples on ice, cooling process has begun

Cooler Temp Read °C: 1.0

Cooler Temp Corrected °C: 0.4

Biological Tissue Frozen?

☐ Yes

☐ No

☒ N/A

Temp should be above freezing to 5°C

Correction Factor: -0.6 °C

Date and Initials of Person Examining Contents:

PK to 11/7/16  
PK

Comments:

Chain of Custody Present?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	1.
Chain of Custody Filled Out?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	2.
Chain of Custody Relinquished?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	3.
Sampler Name and Signature on COC?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	4.
Samples Arrived within Hold Time?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	5.
Short Hold Time Analysis (<72 hr)?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	6.
Rush Turn Around Time Requested?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	7.
Sufficient Volume?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	8.
Correct Containers Used?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	9.
-Pace Containers Used?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	
Containers Intact?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	10.
Filtered Volume Received for Dissolved Tests?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	11. Note if sediment is visible in the dissolved containers.
Sample Labels Match COC?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	12.
-Includes Date/Time/ID/Analysis Matrix: wt.		
All containers needing acid/base preservation will be checked and documented in the pH logbook.	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	See pH log for results and additional preservation documentation
Headspace in Methyl Mercury Container	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	13.
Headspace in VOA Vials (>6mm)?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	14.
Trip Blank Present?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	15.
Trip Blank Custody Seals Present?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	
Pace Trip Blank Lot # (if purchased):		

CLIENT NOTIFICATION/RESOLUTION

Field Data Required? ☐ Yes ☐ No

Person Contacted: \_\_\_\_\_

Date/Time: \_\_\_\_\_

Comments/Resolution: \_\_\_\_\_

ECAL WAIVER ON FILE Y N

TEMPERATURE WAIVER ON FILE Y N

Project Manager Review:

VAP for LMF

Date:

11-3-16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e. out of d, incorrect preservative, out of temp, incorrect containers)